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(NASA-TM-78834) STRENGTH ENHANCEMENT
PROCESS FOR PREALLOYED POWDER SUPERALLOYS
(NASA) 31 p HC A03/MF A01 CSCL 11F

N78-21266

Unclas

G3/26 12413

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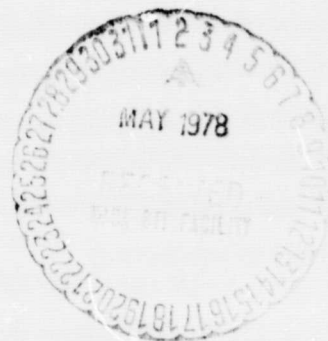
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STRENGTH ENHANCEMENT PROCESS FOR PREALLOYED POWDER SUPERALLOYS

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TECHNICAL PAPER presented at the
1977 TMS-AIME Fall Meeting
Chicago, Illinois, October 24-27, 1977



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ABSTRACT

A technique involving superplastic processing and high pressure autoclaving was applied to a nickel base prealloyed powder alloy. Tensile strengths as high as 2865 MN/m^2 (415 ksi) at 480° C (900° F) were obtained with as-superplastically deformed material. Appropriate treatments yielding materials with high temperature tensile and stress-rupture strengths (980° C (1800° F)) were also devised.

SUMMARY

E-9544 A high temperature, normally cast, stable, nickel base alloy, NASA-TRW VI-A, was made by a prealloyed powder process and subjected to superplastic deformation. Some of the superplastically deformed material was subsequently heat treated and autoclaved. Material strength properties varied widely depending on the material condition. At low temperatures the highest strengths were obtained with the as-superplastically deformed material and at higher temperatures the superplastically deformed and autoclaved material was superior. Autoclaving permitted heat treatments above the alloy's minor phase melting point. In this way significant grain growth as well as microstructural stability could be achieved.

It was also shown how a processing approach might be applied to a nickel base prealloyed powder alloy to achieve ultra-high strengths in the low temperature (480° C (900° F)) range. This approach involves controlled superplastic deformation at 1095° C (2000° F) and resulted in tensile strengths as high as 2865 MN/m^2 (415 ksi) at 480° C (900° F). Aircraft turbine disks fabricated utilizing this technique could offer both substantial strength advantages and fabrication cost reduction over current material/fabrication technology.

Utilizing the superplastic processing approach and combining it with relatively low temperature (980° C (1800° F)) high pressure autoclave treatments can result in material strength variations in an end component that is expected to operate over a broad stress-temperature environment. This processing technique has the potential for uniquely matching material strength capability with design requirements in complex components such as turbine blades.

INTRODUCTION

Superalloy products made from powders have potential advantages relative to castings and conventional forgings. In gas turbine applications, high strength castings are used primarily for high temperature blades and vanes. High strength, high temperature casting alloys usually cannot be mechanically worked, forged or shaped.

Gas turbine blade and nozzle vane alloys with higher use-temperature (and/or strength) capability have been achieved during the last several years by compositional modifications and by innovations in casting methods. To achieve greater strengths at lower temperatures for applications such as gas turbine disks, the fabricators have had, until recent powder metallurgy advances, to think in terms of improving forging alloys.

In both blades and disks, there are areas of differing temperature and stress requirements. Unfortunately, when improvements are made in a material that result in increased high temperature strength (desired in turbine blade airfoils), a decrease usually occurs in its strength and ductility at lower temperatures. This is undesirable for blade applications in that the fir tree and roots of blades which operate at lower temperatures than the airfoils require great strength and appreciable ductility. Typical critical metal temperatures in present day commercial gas turbine blade airfoils usually range from approximately 870° to 980° C (1600° to 1800° F) while those of the fir tree roots are on the order of 650° to 760° C (1200° to 1400° F). Stresses in the blade roots may be three-to-four times as great as those in the critical portions of the airfoils. In the case of gas turbine disks, rim temperatures are maintained at 650° C (1200° F) or lower, but stresses can be several times greater than those in the blade fir trees or roots.

Several of the conventionally nonworkable cast nickel and cobalt base superalloys have been made from pre-alloyed powders and subsequently consolidated by extrusion. Such extrusions not only have been deformable, but they have exhibited exceptionally high strength at the lower temperatures on the order of 650° C (1200° F). However, at higher temperatures of 870° C (1600° F) and above, these extrusions usually have exhibited low strength (refs. 1 through 4). The low strength at high temperatures occurred concurrently with increasing ductility. The extruded prealloyed powder products demonstrated superplasticity at high temperatures. Further, these products were largely in a solid solution state and/or were fine grained. Various heat treatments to cause grain growth and subsequent precipitation hardening, increased the strengths of several of these extruded prealloyed powder alloys considerably. Early work by the authors with autoclaving (refs. 4 and 5) showed that high pressure heat treatments had considerable promise for improving the high temperature strength of such extruded powder metallurgy products.

The NASA-TRW VI-A alloy (refs. 6 and 7) was chosen for this investigation as a material representative of strong, currently used, cast nickel base alloys. In cast form, this alloy has proved to be one of the strongest superalloys developed for gas turbine blade applications. Earlier work (ref. 8) had indicated that a very high tensile strength (1630 MN/m² (236 ksi)

at 650° C (1200° F)) was obtainable when the alloy was made by extruding prealloyed powders. Also, work with VI-A (ref. 8) as well as with other cast nickel base alloys (ref. 4) made by the prealloyed powder process has shown that procedures involving powder extrusion and high pressure autoclave treatments improve high temperature properties over as-extruded and conventionally heat treated powder products. Concurrent with these improvements however, serious degradation of low temperature (650° C (1200° F)) strength occurred.

The present investigation was intended to gain additional insight into the effects of superplastic deformation and autoclaving treatments on the tensile and stress-rupture strengths of the extruded VI A prealloyed powder product. It was hoped that it would be possible to improve the high temperature properties of this prealloyed powder alloy and still maintain high strength at low temperatures. Accomplishment of this goal would suggest that prealloyed powder parts could be tailored to meet differing strength and temperature requirements.

Extruded bars made from prealloyed powders of alloy VI-A were (1) autoclaved at 1315° C (2400° F) for 4 hours (condition A), or (2) superplastically deformed in tension at 1095° C (2000° F) (condition B), or (3) superplastically deformed in tension at 1095° C (2000° F) and autoclaved at 980° C (1800° F) for 4 hours (condition C), or (4) superplastically deformed either in tension or compression at 1095° C (2000° F), heat treated at 1300° C (2375° F) for 1 hour, and autoclaved at 980° C (1800° F) for 4 hours (condition D). Tensile tests were conducted with material so processed at temperatures ranging from room to 1090° C (2000° F) and stress rupture tests at temperatures ranging from 650° to 1095° C (1200° to 2000° F). A metallographic study was made to delineate the microstructural features of the extruded VI-A prealloyed powder product in its various processed forms.

ALLOY PREPARATION

Commercially prepared prealloyed powder of NASA TRW VI-A alloy (same lot as used in ref. 8) was used in the present investigation. The chemical analyses of the VI A alloy powder in the form of ingot, powder and extrusions, as reported in reference 8, are listed in table I along with the composition range that was specified to the vendor for the cast VI-A pig product. Chemistries of the ingot material, powder, and extrusions all agree with the specified nominal compositional ranges for all elements except tungsten. This latter element was slightly lower than the specified ranges in both the powder and extruded forms.

The procedure for preparing the alloy is described in detail in reference 8. Briefly, the as-cast material was vacuum melted and poured into a high pressure argon gas jet that atomized and solidified the metal stream into powder form. The acceptable powder yield (-100 mesh) was about two-thirds of the weight of the total atomized material. About one-third of the atomized material was rejected because of geometry or size considerations (oversized or not spherical).

Table II (taken from ref. 8) lists the particle size distribution of the acceptable atomized powders. The powder was stored under argon gas until it was compacted in the extrusion process.

The -100 mesh powders were chemically analyzed (ref. 8) prior to canning and extrusion. Mild steel cans 7.6 cm (3.0 in.) in diameter by 16 cm (6.3 in.) long were used to contain the powders for the extrusion process. The cans were vacuum degassed after filling (filled while in an argon environment), and after preheating to 260° C (500° F) the cans were sealed by electron beam welding. Each can contained about 2 kg (4.5 lb) of powder. Prior to extrusion, the sealed canned powders were first preheated for 1 hour at 815° C (1500° F) in an explosion proof furnace as a safety check and then soaked for 1 hour at the extrusion temperature of 1190° C (2175° F). A reduction ratio of 17 to 1 was used in the extrusion process. Extrusion dies were fabricated of tool steel and had an included angle of 90°. The extrusion product, including the canning material, was a straight bar of circular cross section with an outside diameter of about 1.9 cm (3/4 in.). In cross section, the extruded bar contained a compacted core of VI-A material about 1.3 cm (1/2 in.) diameter surrounded by the mild steel jacket.

Extrusions were machined to remove the mild steel jacket and sectioned into 7.6 cm (3.0 in.) lengths. These lengths were then either ground to tensile/stress-rupture bars (fig. 1) or used as blanks for additional processing steps.

THERMAL/MECHANICAL TREATMENTS

All material used to make test specimens was produced as described in the preceding section and thus, was initially in the as-extruded condition. Table III lists all treatments applied to the specimens (conditions A to D). Each treatment will be described in more detail in the following sections.

Condition A - High Temperature Autoclaving

The treatment used to produce material in condition A was based on experience obtained in reference 8. It was known from the previous work that the as-extruded VI-A prealloyed powder product was weak at temperatures of 870° C (1600° F) and above. It was also known that to increase the high temperature strength of extruded prealloyed powder products it is necessary to increase the grain size appreciably. The condition A material was subjected to a temperature above the solidus (minor phase melting point) to maximize grain growth. The simultaneous application of pressure in the autoclave was known from previous work to eliminate void formation that would otherwise occur at temperatures above the solidus. The condition A treatment consisted of 4 hours at 1315° C (2400° F) at a pressure of 69 to 105 MN/m² (10 to 15 ksi). The autoclave procedure is described in detail in references 5 and 8 to 11.

Condition B - Superplastic Deformation Treatment

Test specimens of the type shown in figure 1 were elongated in tension at 1095° C (2000° F) approximately 100 to 200 percent by application of a dead weight load that resulted in an initial stress of 28 MN/m² (4 ksi).

Deformation proceeded at a rate of about 0.2/min. The purpose of this treatment was to provide information as to the strength of the VI-A powder product after it had been superplastically deformed to determine if such deformation would act as a strengthening mechanism.

Condition C - Superplastically Deformed

Material Autoclaved at Low Temperatures

Condition C material was obtained by imposing a low temperature autoclave treatment on specimens such as those shown in figure 2. These specimens have been superplastically deformed in tension as described for condition B above. The autoclave treatment was chosen to effectively close internal voids which might have been formed in the microstructure during processing and at the same time retain, as far as possible, the microstructural features of condition B. The treatment consisted of 4 hours at 980° C (1800° F) at a minimum pressure of 69 MN/m² (10 ksi).

Condition D - Superplastically Deformed Material Subjected to High
Temperature Heat Treatment Plus Low Temperature Autoclaving

Specimens of the type shown in figure 2 were superplastically deformed in tension prior to applying a high temperature (1300° C (2375° F)) heat treatment followed by a low temperature (980° C (1800° F)) autoclaving treatment at 69 to 275 MN/m² (10 to 40 ksi). The specimens were superplastically deformed in tension as described for condition B. The initial 1300° C (2375° F) heat treatment was applied to increase grain size and thereby the high temperature strength potential of the material. The low temperature autoclaving was applied to close voids in the microstructure.

Other specimens of the types shown in figure 3 were ground from material superplastically deformed in compression. This was done after giving them the heat treatments and autoclaving indicated above for the condition D tensile deformed specimens. Sub sized specimens with dimensions as shown in figure 3 were used because the compression deformed blanks approached sheet thickness. The steps in making the specimens shown in figure 3 were as follows: Flats were ground on the tapered ends of machined extruded specimens (fig. 4(a)) to form uniformly thick bars. These bars were placed between flat dies, heated and pressed. Compression deformation occurred at 1095° C (2000° F) under a stress of approximately 20 MN/m² (3 ksi). The bar was allowed to deform until it was reduced to a flat about one-third of its original thickness (fig. 4(b)). After heat treating and autoclaving specimens of type A and B (fig. 3) were then machined from these flats.

A limited number of tests with compression deformed specimens were run to determine if the results would compare closely with those obtained for the tensile deformed specimens of condition D. It was necessary to make this comparison because most parts or components will probably be produced by compressive deformation processes such as pressing or creep forming.

GRADIENT HEAT TREATMENT STUDIES

Special metallographic specimens were prepared for a gradient heat treatment study. An as-extruded bar approximately 7.6x1.5 cm (3x0.6 in.) in diameter was placed between flat platens and superplastically deformed at 1095° C (2000° F) to a thickness of about 0.5 cm (0.2 in.). The length of the specimen remained approximately the same. Heat treating was done in a gradient furnace. One end of the specimen was heated to 1315° C (2400° F) and the other to 1095° C (2000° F). Thermocouples attached to the specimens indicated that a uniform temperature gradient existed between the ends of the specimen. After the application of the gradient heat treatment, the specimen was sectioned lengthwise. One portion was given a low temperature autoclaving treatment at 980° C (1800° F) and 173 MN/m² (25 ksi) for 1 hour, the other was not. The sections were compared metallographically to determine the effects of the low temperature autoclaving treatment on the microstructures.

GENERAL METALLOGRAPHY

Representative samples of the VI-A alloy in the various conditions investigated were examined metallographically. Samples were prepared by sectioning, mounting, and mechanically polishing. They were etched in a solution of 33 parts water, 33 parts acetic acid, 33 parts nitric acid, and 1 part hydrofluoric acid. Specimens were examined under a light microscope at magnifications up to 750x.

RESULTS AND DISCUSSION

The following sections describe results obtained from both mechanical property determinations and metallographic studies.

Tensile Properties

All tensile data are listed in table IV. Average tensile strengths are plotted in figure 5 as a function of temperature for all conditions of VI-A investigated. In addition, as-extruded and as-cast tensile curves from reference 8 are included for comparison. Strength and ductility values vary widely over the test temperature range investigated for conditions A, B, C, and D. Tensile properties generally exhibited the expected trend of continuously decreasing strength as temperature was increased above room temperature. The one major exception to this was condition B which represented the as-superplastically deformed material. It exhibited extremely high strength values at 480° C (900° F) with ultimate tensile strengths as high as 2865 MN/m² (415 ksi). The average strength of this material was 3210 MN/m² (335 ksi) at 480° C (900° F). This is an increase of 700 MN/m² (100 ksi) over room temperature strength. The tensile strengths of the superplastically deformed materials that were not subsequently subjected to high temperature heat treatments (conditions B and C) also had higher strengths at room temperature and 650° C (1200° F) than those that were heat treated at the higher temperatures (conditions A and D). The

tensile strength of the material that had been heat treated at high temperatures (conditions A and D) was superior at 980°C (1800°F) to that of material not subjected to high temperature heat treatment (conditions B and C).

For greater ease of comparison, the tensile properties of material in all the conditions investigated are shown in figures 6 and 7 for 650°C (1200°F) and 980°C (1800°F), respectively. Also shown are the as-extruded powder product properties and the as-cast alloy properties (ref. 8). These figures will be referred to in some detail in a subsequent section of this report that deals with potential applications.

Stress Rupture Properties

Stress rupture data are listed in table V. Larson-Miller type plots for each material condition investigated are shown individually in figures 8(a) through (d) and are summarized for each comparison in figure 8(e). It was assumed that the tensile strengths obtained were approximately equivalent to a 0.1 hour stress rupture life. This was done to better define the Larson-Miller curve over a broader stress and temperature range. It should be noted from figure 8(d) that the data obtained for material superplastically deformed in compression had a stress rupture life very close to that of material superplastically deformed in tension after being subjected to the condition D treatment.

Of the materials studied, the as-superplastically deformed material (condition B), had the highest stress rupture strengths up to temperatures of approximately 650°C (1200°F). Beyond this point, the rupture strength fell off rapidly and tests were not made at temperatures above 870°C (1600°F). The superplastically deformed material which was subjected to a low temperature autoclaving treatment (condition C) had properties that were considerably lower than the as-superplastically deformed (condition B) material over the entire temperature range studied. This was in contrast to what was observed in the tensile tests (fig. 5) where the condition C material had nearly equivalent properties to those of the condition B material above 650°C (1200°F). At temperatures above approximately 705°C (1300°F) the material subjected to the high temperature heat treatments (conditions A and D) had very similar stress-rupture properties. The condition D material exhibited high stress-rupture properties at both low and high temperatures. For example, from figure 8(d) it can be shown to have a 300 hours stress rupture life at both 650°C (1200°F) and 1035 MN/m^2 (150 ksi) and at 980°C (1800°F) and 105 MN/m^2 (15 ksi). Thus, it is apparent that the combination of superplastic deformation, ambient and autoclave heat treatments can produce a material that has potential for use over a wide temperature range.

To provide a common basis for comparing the different forms of VI-A, the Larson-Miller plots of figures 8(a) through (d) were used to obtain 100 hour use-temperatures. These are shown in figure 9 together with the use-temperatures for the as-cast and as-extruded powder products at stress levels of 105, 345, 690, and 1035 MN/m^2 (15, 50, 100, and 150 ksi).

The use temperatures for material subjected to the high temperature heat treatments (conditions A and D) were similar at all but the highest stress level. At the highest stress level the combination of superplastic deformation together with ambient and autoclave heat treatments (condition D) had a 80°C (150°F) use temperature advantage over condition A (675°C (1250°F) vs 595°C (1100°F)). The material in condition D was superior or equivalent in use temperature to the as-extruded product at all stresses. However, compared to the as-cast alloy, condition D material was superior at only the two highest stress levels (e.g., 110°C (200°F) at 1035 MN/m^2 (150 ksi) and 10°C (20°F) at 690 MN/m^2 (100 ksi)). At the lowest stress level (105 MN/m^2 (15 ksi)) it had a 55°C (100°F) lower use temperature.

In figure 10, the Larson-Miller plots for conditions A, B, C, and D are shown as a shaded band. As-extruded powder VI-A, cast VI-A, and one of the strongest directionally solidified alloys, DS Mar M-200+Hf, are also shown. At high stresses and corresponding low temperatures, the band encompasses the as-cast and as-extruded powder products. At lower stress levels and corresponding high temperatures, the band also encompasses the as-extruded product but falls below that of both cast alloys. It is possible that treatments similar to those used in this investigation with VI-A, when applied to other strong nickel base alloys, might produce materials more comparable in strength at the high temperatures to the directionally solidified Mar M-200+Hf alloy. This possibility is worth investigation.

Metallography

Microstructures of atomized powders are shown at 250x, 750x, and 26 000x in figure 11 taken from reference 8. These powders are spherical in appearance and contain what appears to be microporosity or entrapped gas (argon from the atomization process) pockets. Each powder particle appears to be composed of solid solution grains with no precipitates in evidence. The boundaries contain carbides and gamma prime. The diameter of the grains within the powder particles was about 0.009 mm (0.004 in.). This corresponds to ASTM grain size number 11.

The microstructure of the as-extruded powder product at 750x and 26 000x is shown in figure 12 (ref. 8). The grain structure appears to have been refined by the extrusion process. Both carbide and γ' precipitation can be observed (fig. 12(b)). The grain boundaries are indistinct and the diameter of the grains was reduced by extrusion to about 0.0014 mm (0.00005 in.), ASTM grain size 16.

Figure 13 shows the microstructure of the condition A material at 250x and 750x. In this condition, the grain size increased compared to the as-extruded product to about 0.1 mm (0.004 in.). Large islands of primary gamma prime formed at the grain boundary interfaces. Discontinuous chains of gamma prime are apparent in the grain boundary areas and gamma prime particles of about 0.005 mm (0.0002 in.) are distributed throughout the matrix. The concentrations of large gamma prime particles suggest that this is not an optimum microstructure for achieving either maximum strength or ductility, especially in the intermediate temperature range.

Microstructures of condition B material are shown in figure 14 at 250x and 750x. As might be expected, there is a marked similarity between condition B material and the as-extruded powder product, since the superplastic deformation occurred at a temperature less than the extrusion temperature.

Figure 15 shows the microstructure of condition C material at 250x and 750x. Gamma prime is just beginning to form. The gamma prime islands are not yet distinct from the matrix. Grain boundaries are not evident and the fine gamma prime particles are much finer than those observed for material in condition A. These fine (secondary) gamma prime particles are less than 0.0025 mm (0.0001 in.) in diameter.

Figure 16 shows the microstructure of condition D material at 250x and 750x. Gamma prime appears as discontinuous segments in the grain boundary regions along with some carbides. Carbides are also evident in the matrix. The gamma prime cells in the matrix are less than 0.0025 mm (0.0001 in.) in diameter, as are the carbides. The gamma prime has not agglomerated to any significant degree. Material in condition D has a grain size of ASTM 6 to 7.

The largest grain size in any of the four conditions of the VI-A prealloyed powder product that were considered was observed in condition A material. Condition A material also showed the largest amount of agglomeration of gamma prime as well as the largest size gamma prime precipitates. Condition A material appears to be overly coarse for optimum properties. Condition D material on the other hand, has a smaller grain size and a more uniform distribution of the gamma prime phase than condition A material. The smaller grain size would suggest that better low temperature strength properties could be obtained than would be possible with the larger grained material of condition A. Also, the high temperature strength would normally be expected to be lower than that of condition A material because of the smaller grain size. However, the better distribution of the gamma prime phase in condition D material probably compensates for the smaller grain size and this contributes to the good high temperature strength observed for material in condition D.

Gradient Heat Treatment Studies

Figure 17 shows the microstructure of a compression deformed bar. The upper series of microstructures show changes in structure as a function of exposure temperature from 1090° to 1315° C (2000° to 2400° F) in a gradient heat treating furnace. Agglomeration of gamma prime is not evident until exposure temperature reaches 1200° C (2200° F). The agglomeration continues as the temperature increases up to 1260° C (2300° F) (not shown). Above this temperature, the gamma prime appears to be resolutioned, but the grains continue to grow with increasing temperature. At 1315° C (2400° F) melting with resultant porosity becomes evident. The lower series of microstructures in this figure show the effect of superimposing a 980° C (1800° F) autoclave treatment at 175 MN/m² (25 ksi) for 4 hours on the same gradient heat treated bar. The microstructures are similar in all cases except at the highest temperature. The voids formed by melting have been closed by the autoclaving treatment. It is apparent from the figure that superposition of the autoclaving treatment

on a gradient heat treated powder product component can provide void free microstructures and a large grain size, whereas ambient heat treatments alone intended to achieve comparable grain sizes would result in lack of structural integrity. A variety of grain structures and probably a corresponding variety of strengths could be obtained with a part heat treated and autoclaved utilizing the gradient technique. Such variations in strength with structure were demonstrated in references 4, and 8 through 11 by the authors with a number of prealloyed superalloy powder products.

POTENTIAL APPLICATIONS

In this investigation the VI-A alloy was selected as the vehicle to demonstrate how a normally nonworkable material can be deformed superplastically and then treated to achieve a wide variety of properties. To illustrate the potential of this approach for producing gas turbine components, the alloy properties and behavior already shown will be re-examined. Some aircraft gas turbine criteria will be used as a basis for the subsequent discussion.

Turbine Disks

In turbine disks, both hub and rim requirements must be considered. The latter operates at temperatures on the order of 650°C (1200°F), the former at temperatures on the order of 480°C (900°F). Both tensile and stress rupture properties are of major concern at the rim, and tensile properties (to achieve high burst strength) are of particular concern at the hub. From a tensile standpoint, each condition considered herein has produced a material with tensile strength at 650°C (1200°F) adequate for disk application. Conditions A, B, and C provided materials with adequate ductility as well. Since condition A does not involve superplastic deformation to a shape it is not a likely candidate for disk fabrication. From a stress rupture standpoint, condition B material (as superplastically deformed) is superior to that of condition C. Therefore, on the basis of both tensile and stress rupture properties, condition B material would be the most favorable for meeting disk rim requirements. Referring to figure 5, it should be noted that condition B material also showed outstanding tensile strength at 480°C (900°F), the temperature region of interest for the hub. In view of these favorable tensile and stress rupture properties, condition B material would be the most appropriate for turbine disk application. Thus, turbine disks might readily be fabricated directly to shape by the superplastic deformation of extruded prealloyed powder superalloys such as VI-A. Such disks should have strength advantages over those made by current production methods.

From the author's experience it is known that superplastic deformation of extruded prealloyed powder alloys can produce parts with extremely fine detail and close tolerances. This has been accomplished with low unit loading. As a consequence, reduced disk fabrication costs could be expected since the massive forging facilities required for present day disk forging would not be needed with this approach. Die cost and wear would be significantly reduced, since forming to shape would be achieved without requiring

the normal step forging operations. An example of the degree of deformation that may be achieved with the VI-A extruded product is illustrated in figure 18. A reduction of 87 percent was achieved at 1095°C (2000°F) under a nominal applied load of 17 MN/m^2 (2.5 ksi). It might also be noted that the superplastically deformed sample of figure 18 approaches sheet thickness, having a thickness of 1.5 mm (0.060 in.). This suggests the further possibility of achieving ultra high strength sheet material via this process.

Turbine Blades

A major property criterion for determining the suitability of a material for gas turbine blade application is its stress rupture strength. Figure 19 shows a sketch of a turbine blade and the generalized stress and temperature distribution present. The stress is highest at the root section and decreases along the span. The temperature distribution varies in the opposite manner. The actual values of the stresses and temperatures can vary widely depending upon blade design and engine operating conditions.

As shown in figure 9, the 100 hour stress-rupture use temperature of the superplastically deformed heat treated/autoclaved (condition D) material was greater than that of the cast alloy below 760°C (1400°F) and less above 870°C (1600°F). Although the stress-rupture properties of the prealloyed powder product were not greater than those of the cast version of the alloy over the entire temperature range, the prealloyed powder concept must be considered as having potential for blade applications. Only limited attempts were made in this study to optimize the treatments applied, so as to achieve the ultimate in strength over the entire temperature range. As is discussed in a later section further consideration of a number of variables associated with the overall process remain to be investigated. One might reasonably expect, in view of the significant improvements already shown over the as-extruded powder product, that the results of such work could significantly affect the strength capabilities of prealloyed powder alloys such as VI-A. If this can be accomplished there is a possibility of making turbine blades (process described in a later section) in a more cost effective manner. Cost savings could be achieved with respect to the capital equipment, raw materials, and energy requirements.

POSSIBLE PROCESSING PROCEDURES TO ACHIEVE STRENGTH VARIATIONS IN STRUCTURAL COMPONENTS

In proposing the following it is assumed that compression and tensile superplastic deformation will yield products that have similar strengths. Of course, this assumption is based on limited data for the superplastically compression deformed materials.

It would be desirable to be able to produce different properties in different portions of actual components. Such a multi-property component was described in the previous section; namely, a turbine blade that had different requirements in the root and the airfoil. This investigation has shown that a superplastically deformed powder part can be treated so as to

have differing properties along its axis. For example, a blade that would match the differing stress and temperature along its span could be produced by the procedure described below. The fabrication steps involved will be better understood with the aid of the schematic diagrams of figure 20. To produce the bar stock which is to be superplastically deformed to a blade shape, a similar procedure to that used in this investigation is proposed. This involves canning atomized powders and extruding them to bar stock. The bar stock would then be superplastically deformed to the desired blade shape. The blade would be heat treated in a gradient furnace at ambient pressure. This would produce large grains and high strength where it is needed in the airfoil, while the root and fir tree section would be maintained at a low temperature to preserve the superplastically deformed structure and retain the high strength needed in the cooler blade root.

As shown in this investigation, to achieve the large grains needed for high temperature strength it would be necessary to heat to temperatures that produce melting and void formations. To close these voids the entire blade would then be given a suitable autoclave treatment. It should be noted this procedure would be applicable to other components as well, which might require strength properties that differed at different sections.

CONSIDERATIONS FOR FUTURE WORK

More work is required to fully determine the effects of superplastic deformation in compression. Thus, the effects of temperature of deformation, rates of deformation, degrees of deformation (i.e., total flow of metal in die), orientation of deformed structures (i.e., strengths in direction of flow vs strengths transverse to direction of flow) are only a few of the variables associated with superplastic deformation in dies which should be investigated.

Autoclave temperature is of major importance and it should be noted that the condition D heat treatment involved a relatively low autoclave temperature (980°C (1800°F)). This was found to be sufficiently high to permit void closure at the autoclave pressures available. This autoclave temperature did not affect the alloy's low temperature properties as adversely as did condition A which used a 1315°C (2400°F) autoclave temperature. This is also reflected in the microstructure of the material in condition D which is less coarse than that of condition A (see figs. 13 and 16). Thus, in making a choice of autoclave conditions it is important to strike a proper balance between the temperature and pressure applied to maximize both the high and low temperature properties of the part. Only a limited attempt was made in this investigation to arrive at the most appropriate set of autoclave conditions for the prealloyed powder product considered. Further study to determine the most desirable combinations of autoclave pressure and temperature for nickel base prealloyed powder products would be desirable.

SUMMARY OF RESULTS AND CONCLUSIONS

The following major results and conclusions were obtained from this investigation which applied superplastic deformation and autoclaving treatments to prealloyed powder of the NASA - TRW VI-A alloy.

1. Superplastic deformation of extruded bars made from atomized powder products yielded materials with unusually high strengths at low temperatures. For example, average tensile strengths of the as-superplastically deformed (condition B) material of 2310 MN/m² at 480° C (335 ksi at 900° F) and 1380 MN/m² at 650° C (200 ksi at 1200° F) with reduction of areas of 12 percent or greater were obtained. Stress rupture life at 650° C and 1035 MN/m² (1200° F and 150 ksi) for the as-superplastically deformed material was over 3000 hours with a 9 percent reduction of area.

2. To achieve significant grain growth in the superplastically deformed prealloyed powder product it was necessary to heat treat above the minor phase melting temperature.

3. Autoclaving permitted heat treating of the prealloyed powder material at temperatures above the incipient melting point without structural degradation.

4. As a result of the high temperature heat treatments, high temperature properties were significantly improved. For example, tensile strength was increased from 93 MN/m² at 980° C (13.5 ksi at 1800° F) for the as-extruded material to 490 MN/m² at 980° C (70 ksi at 1800° F) with the high temperature/autoclave treatment (condition D) material. In stress rupture the 105 MN/m² (15 ksi) 100 hour use temperature was increased from 840° C (1540° F) to 1015° C (1860° F).

5. The high temperature heat treatment followed by a low temperature autoclave treatment (condition D) not only yielded a material with good stress-rupture properties at high temperatures, but also did not destroy the strength of the material at the low temperatures (650° C (1200° F)). For example, stress rupture lives of approximately 300 hours were obtained at 980° C and 105 MN/m² (1800° F and 15 ksi) and at 650° C (1200° F) and 1035 MN/m² (150 ksi) for the condition D material.

6. Analyses of the results suggested that a component such as a gas turbine disk, requiring high strength in the low temperature range (480° to 650° C, 900° to 1200° F) might be fabricated by superplastically deforming an as-extruded prealloyed atomized powder product. The tensile and stress-rupture strength of superplastically deformed VI-A appeared to be more than adequate for disk applications. Other superalloys of this type may be expected to respond in a similar fashion.

7. A processing approach is proposed for turbine blades which offers the potential of providing variations in strength which match the differing stress and temperature requirements along the blade span.

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TABLE I. - CHEMICAL ANALYSES OF VI-A POWDER PRODUCTS

Element	Specified composition range	Ingot ^a	Powder ^b	Extrusion ^b
	Weight percent of element			
Tantalum	3.5 to 9.5	9.0	8.68	8.81
Columbium	0.25 to 0.75	.47	.56	.53
Zirconium	0.05 to 0.15	.10	.10	.12
Hafnium	0.20 to 0.70	.41	.48	.50
Rhenium	0.10 to 0.70	.41	.40	.34
Tungsten	5.3 to 6.3	5.92	5.24	5.04
Cobalt	6.5 to 8.5	7.52	7.78	7.58
Titanium	0.75 to 1.25	.97	1.00	.90
Molybdenum	1.5 to 2.5	1.98	1.72	2.17
Chromium	5.6 to 6.6	6.20	5.94	5.86
Carbon	0.08 to 0.18	.15	.140	.141
Silicon	-----	<10	.05	-----
Manganese	-----	<.02	.004	-----
Iron	-----	-----	.06	-----
Sulfur	-----	.005	10 ppm	-----
Aluminum	5.0 to 5.8	5.53	5.41	5.25
Boron	0.012 to 0.024	-----	-----	-----
Nickel	Balance	Balance	Balance	Balance
Oxygen	-----	-----	90 ppm	59 ppm

TABLE II. - PARTICLE SIZE DISTRIBUTION OF ALLOY

VI-A ATOMIZED POWDER^a

[Screen analysis.]

Mesh opening, mm square	Tyler screen size	Particle size distribution, percent
>.210	>65	0
.210/.149	65/100	1.0
.149/.105	100/150	9.7
.105/.074	150/200	17.0
.074/.053	200/270	14.2
.053/.037	270/400	14.9
<.037	<400	43.2

^aAs reported by vendor.

TABLE III. - THERMAL/MECHANICAL TREATMENTS APPLIED TO VI-A POWDER PRODUCT

PROCESSING STEPS						
Condi- tion	Deformed at 1095° C (2000° F) in		Heat treated 1 hour at 1300° C (2375° F)	Autoclave 4 hour at 980° C (1800° F) 69 to 275 MN/m ² (10 to 40 ksi)	Autoclave 4 hour at 1315° C (2400° F) 69 to 105 MN/m ² (10 to 15 ksi)	Grain size, ASTM
	Tension	Compression				
A	✓				✓	4-5
B	✓			✓		16
C	✓		✓	✓		16
D	✓	✓				6-7

TABLE IV. - TENSILE DATA

Condition	Test temperature		Ultimate tensile strength		Elongation, percent	Reduction of area, percent
	°C	°F				
			MN/m ²	ksi		
A	20	70	1250	180.5	13	12
	650	1200	1140	165.5	9	11
	650	1200	1230	179.0	17	--
	760	1400	980	141.5	3	14
	760	1400	1070	155.0	8	--
	980	1800	480	70.0	5	--
	980	1800	460	67.0	4	4
	1095	2000	225	32.5	4	--
	1095	2000	260	37.5	3	--
B	20	70	1910	227.0	*	16
	480	900	2865	415.0	>7	14
	480	900	1965	285.0	*	12
	480	900	2105	305.0	*	13
	650	1200	1380	200.0	*	12
	870	1600	520	75.0	*	45
	980	1800	290	42.0	*	--
C	20	70	1510	219.0	*	10
	650	1200	1450	210.0	*	11
	980	1800	155	22.6	*	85
D	20	70	1175	170.0	*	6
	480	900	1455	211.0	*	6
	650	1200	1035	150.0	*	1
	650	1200	1090	158.0	*	1
	980	1800	520	75.0	*	--
	980	1800 ^a	460	67.0	6	3

*Nonuniform diameter test section, due to tensile deformation processing.

^aCompression deformed bar (fig. 3) type B.

TABLE V. - STRESS RUPTURE DATA

Condition	Stress		Temperature		Life, hr	Elongation, percent	Reduction of area, percent
	MN/m ²	psi x10 ⁻³	°C	°F			
A	1035	150	650	1200	2.8	6	--
	1035	150	650	1200	2.8	7	--
	760	110	650	1200	306.8	2	--
	690	100	760	1400	44.3	1	--
	545	79	760	1400	3409.6	4	4
	415	60	870	1600	22.3	2	--
	275	40	870	1600	322.0	5	--
	205	30	980	1800	12.0	2	--
	110	16	980	1800	287.0	10	--
	52	7.5	980	1800	2811.2	12	6
B	1035	150	650	1200	3097.5	*	9
	690	100	760	1400	5.3	*	27
	275	40	870	1600	1.0	*	--
	940	136	675	1250	651.8	*	19
C	1035	150	650	1200	108.6	*	7
	545	79	760	1400	7.3	*	19
D (Tensile deformed)	1035	150	650	1200	264.1	*	4
	690	100	760	1400	64.0	3	--
	690	100	760	1400	41.4	*	--
	620	90	760	1400	253.5	*	--
	275	40	870	1600	125.5	*	1
	275	40	870	1600	64.7	*	4
	275	40	870	1600	107.4	*	1
	220	32	980	1800	93.6	*	2
	110	16	980	1800	386.7	*	9
	110	16	980	1800	81.3	*	2
	55	8	1095	2000	3.2	*	23
D (Compression deformed)	1035	150	650	1200	287.6 ^a	--	--
	220	32	980	1800	10.0 ^a	--	--
	275	40	870	1600	60.2 ^b	--	--
	110	16	1035	1900	3.0 ^b	30	15

^a Specimen was run for 287.6 hr at 650° C/1035 MN/m² (1200° F/150 ksi), then rerun at 980° C/220 MN/m² (1800° F/32 ksi) to failure (i.e., 10 hr) type A specimen.

^b Type B specimen.

* Nonuniform diameter test section, due to tensile deformation processing.

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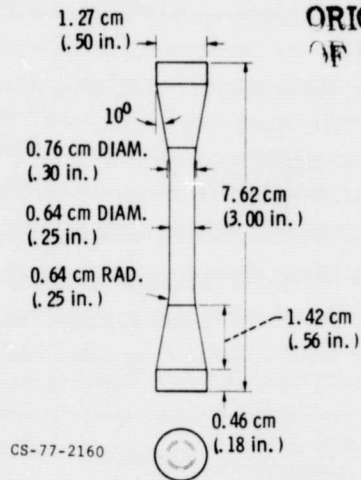


Figure 1. - Tensile/stress rupture specimen ground from extruded bar stock (used in condition A).



Figure 2. - Typical tensile deformed tensile/stress-rupture specimens tested in conditions B, C, and D.

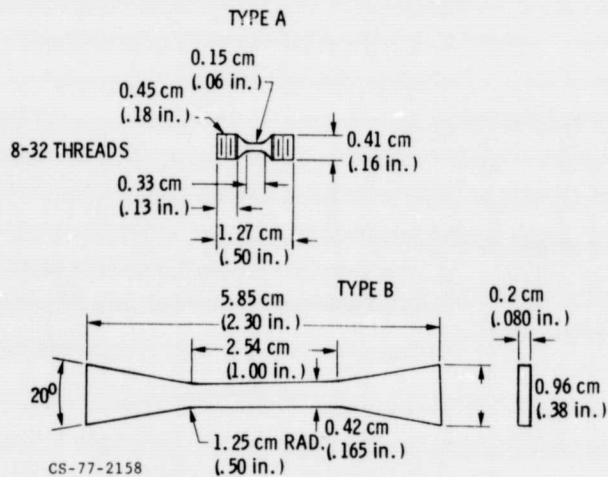
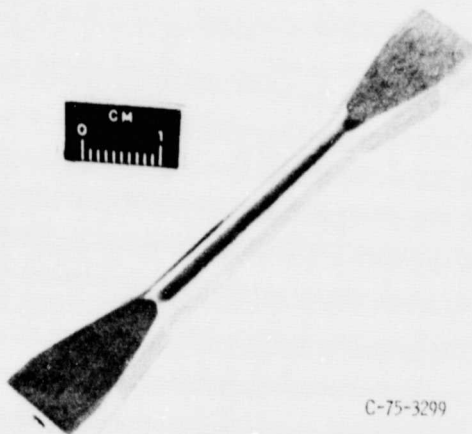
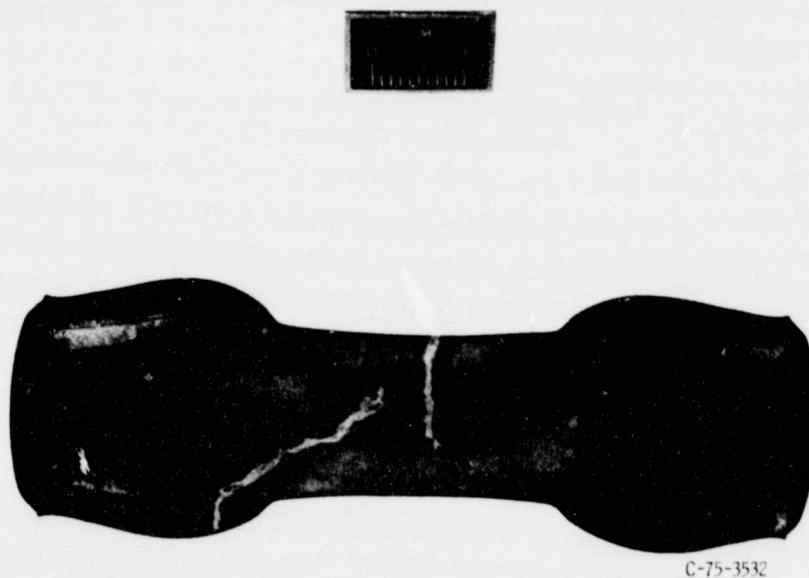


Figure 3. - Compression deformed sub sized specimens used in condition D tests.



(a) TENSILE/STRESS-RUPTURE BAR PRIOR TO COMPRESSION DEFORMATION

Figure 4. - Compression test bar processing.



(b) TENSILE/STRESS-RUPTURE BAR AFTER COMPRESSION DEFORMATION AT 1090°C (2000°F).

Figure 4. - Concluded.

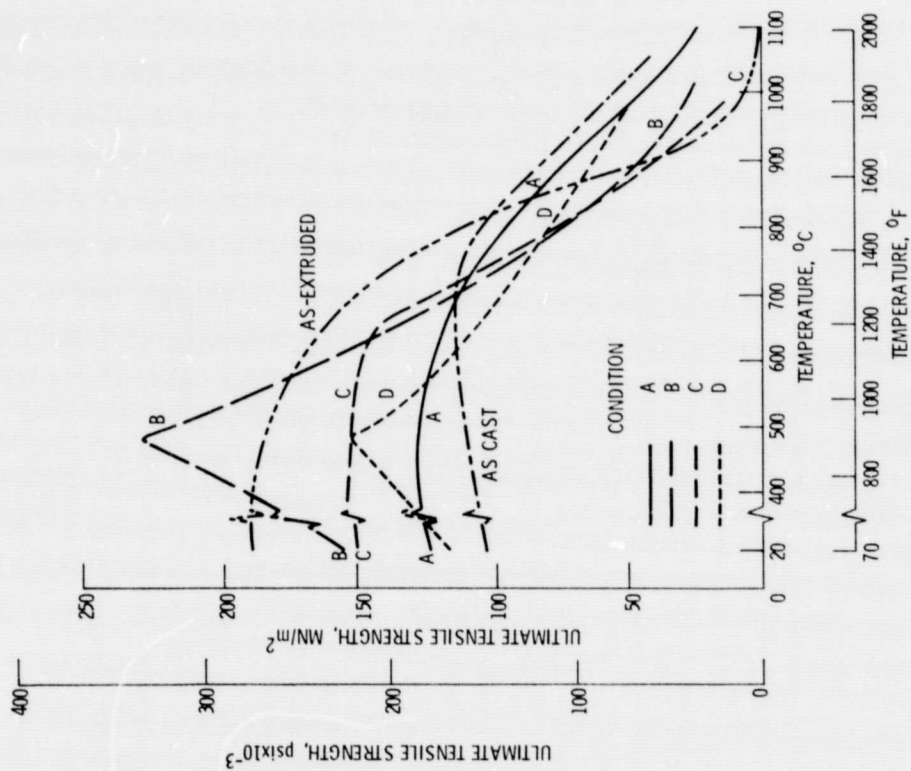


Figure 5. - Tensile strength of VIA in several conditions.

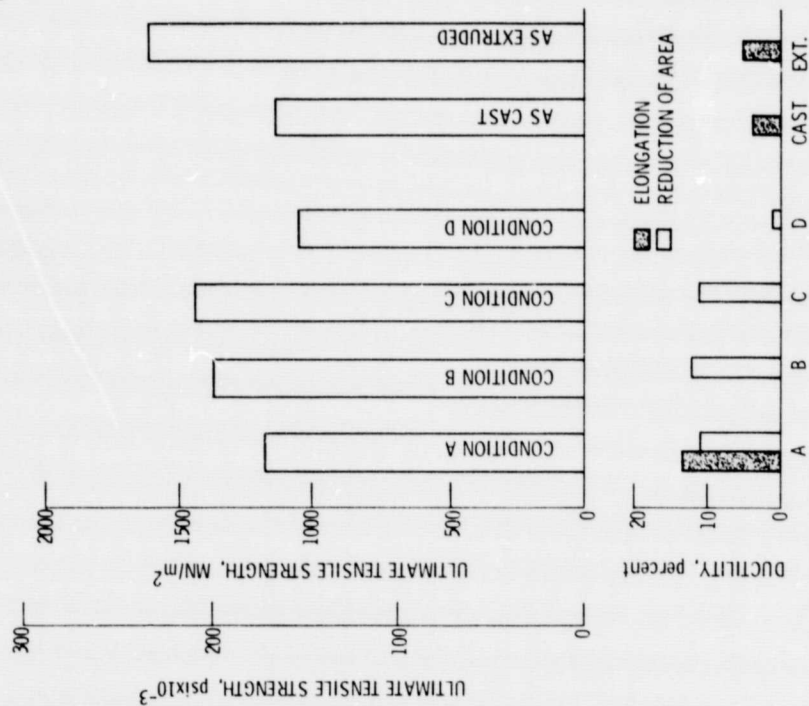


Figure 6. - Tensile properties at 620° C (1200° F) of VIA in various conditions.

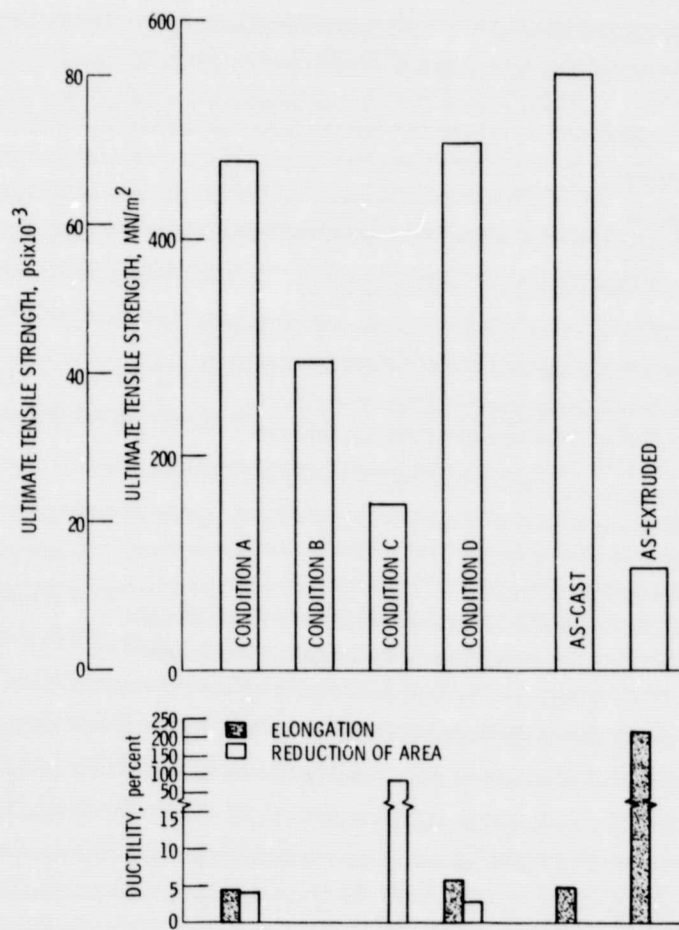


Figure 7. - Tensile properties at 980° C (1800° F) of VIA in various conditions.

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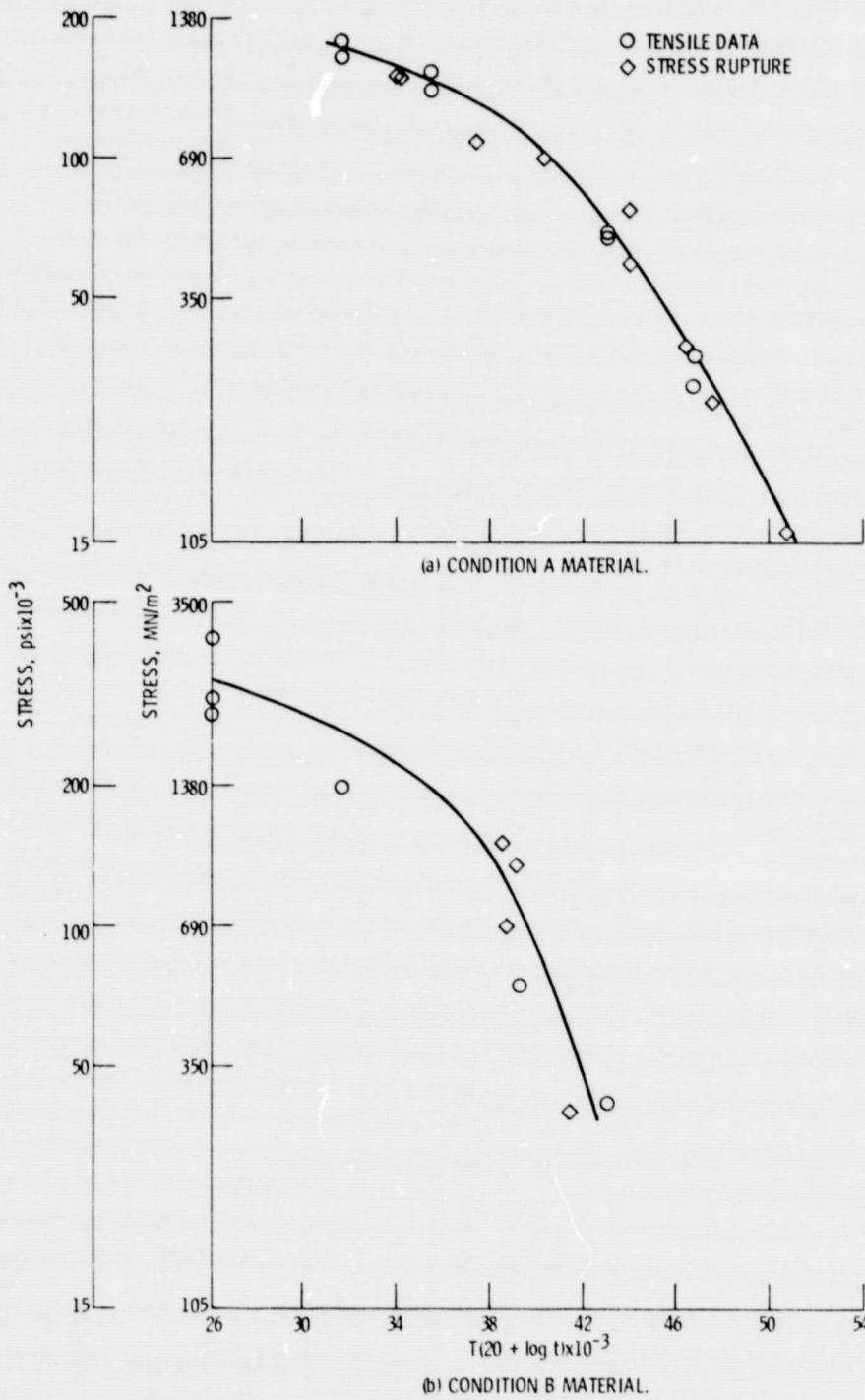


Figure 8. - Larson-Miller plot of VIA prealloyed powder product in several conditions.

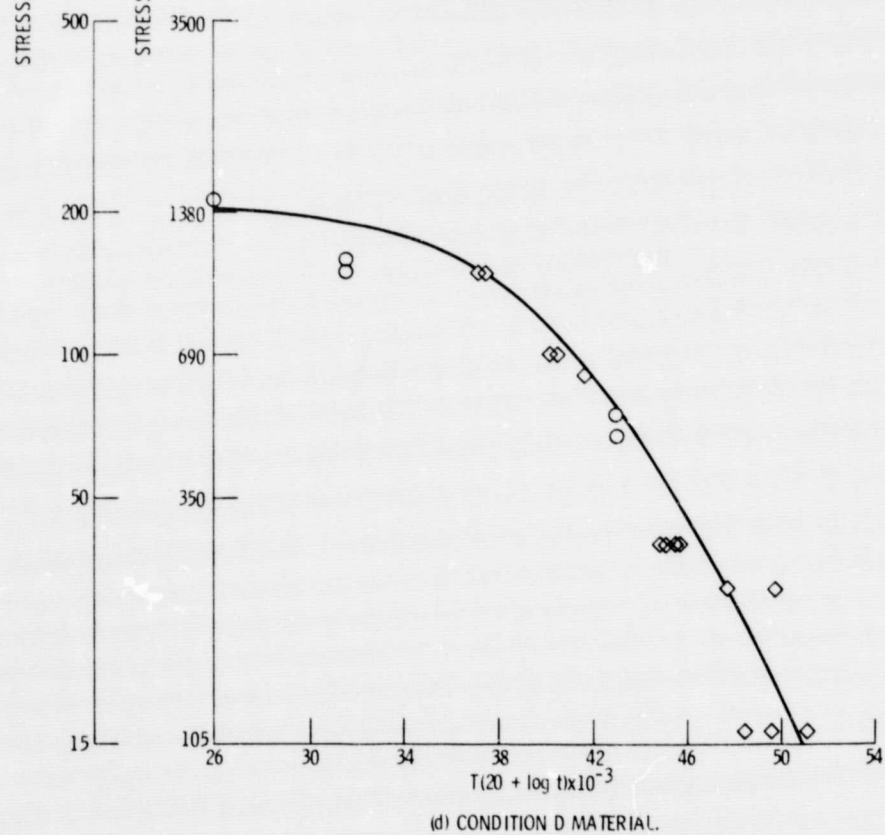
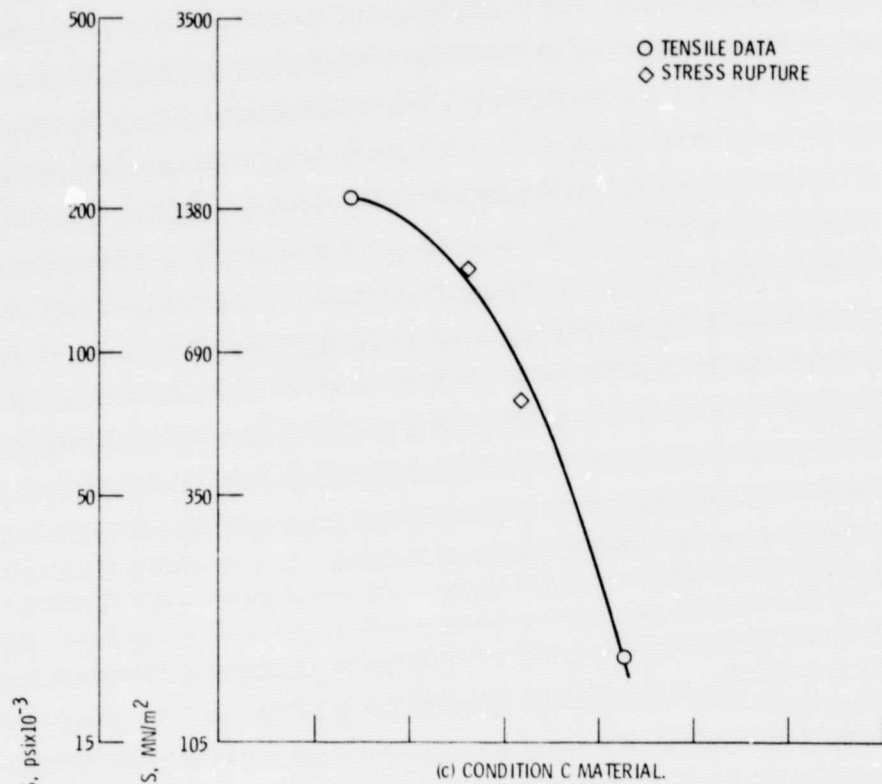
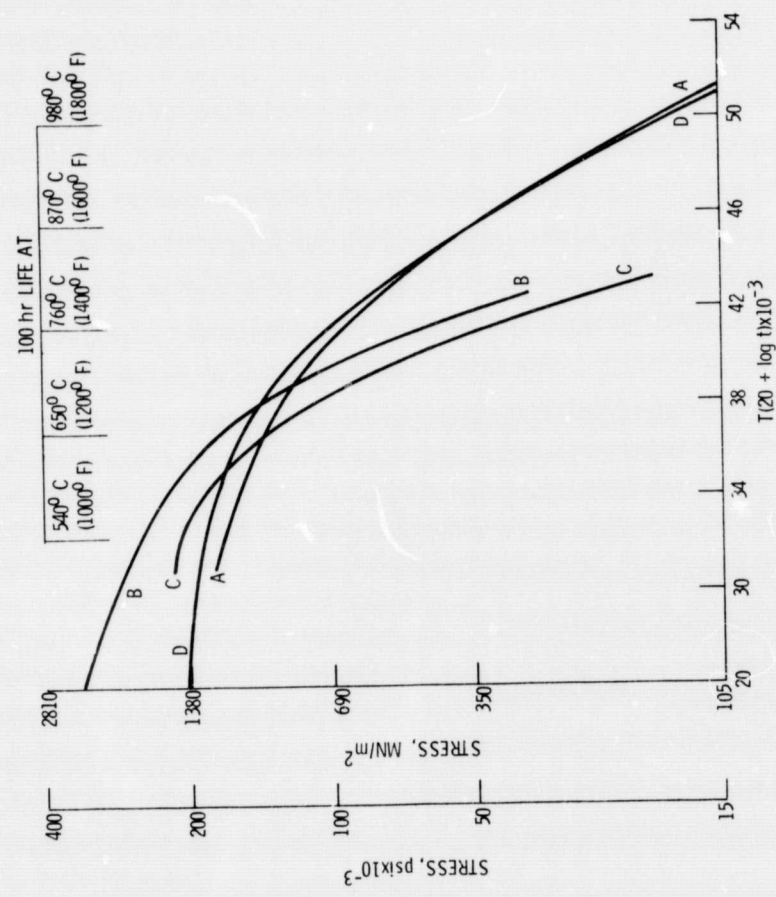


Figure 8. - Continued.

- TEST STRESSES
- 1 105 MN/m² (15 000 psi)
 - 2 345 MN/m² (50 000 psi)
 - 3 690 MN/m² (100 000 psi)
 - 4 1035 MN/m² (150 000 psi)



(e) ALLOY CONDITIONS COMPARED.

Figure 8. - Concluded.

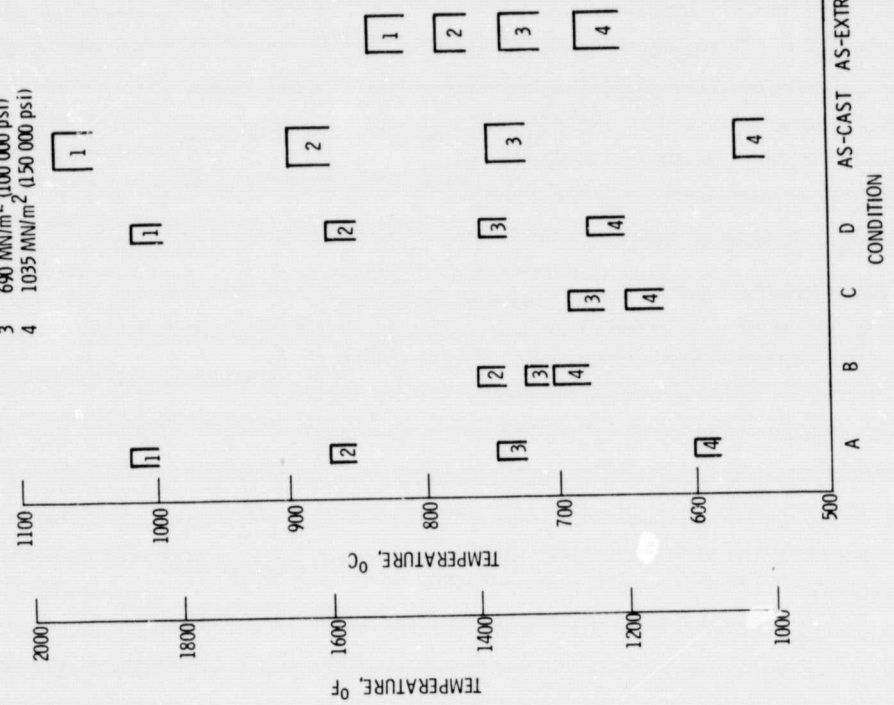


Figure 9. - 100 Hour use temperature of VIA in several conditions.

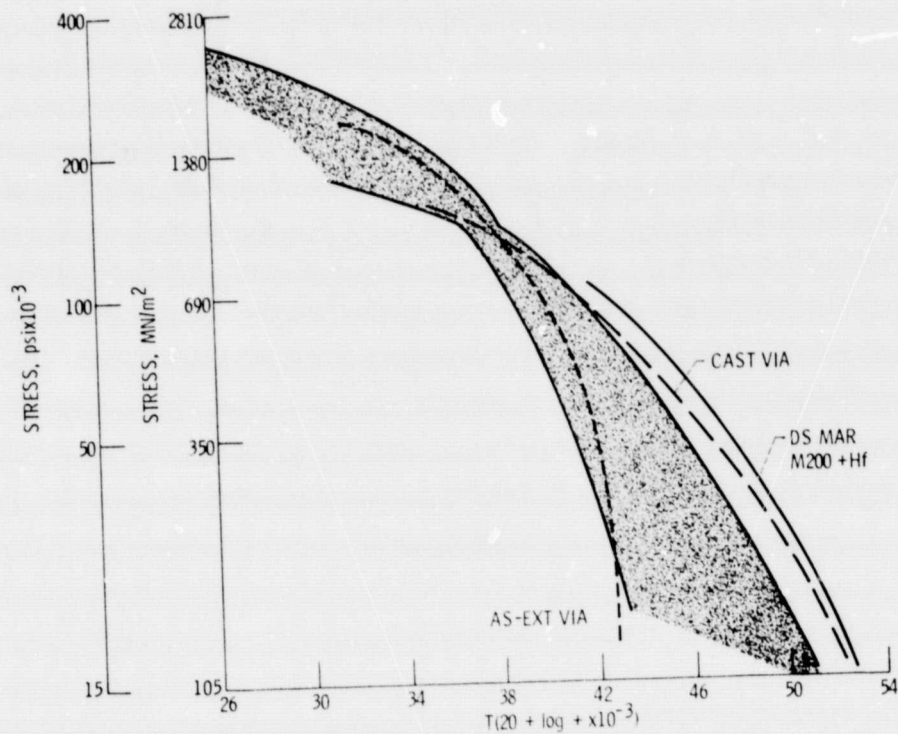


Figure 10. - Strength property range of VIA in conditions A through D compared to other materials/forms.

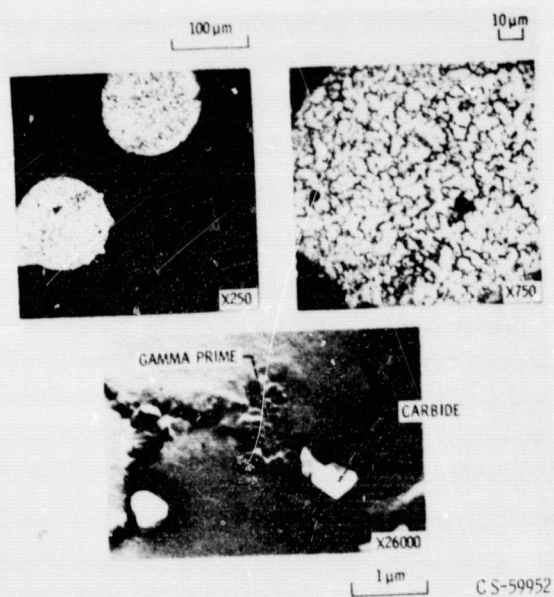


Figure 11. - As-received powders of VI A alloy.

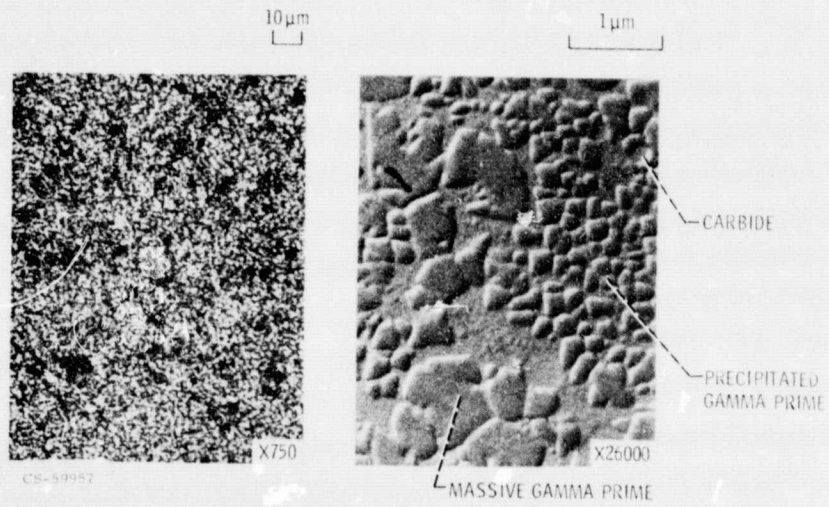


Figure 12. - As-extruded powder product of VI A alloy.

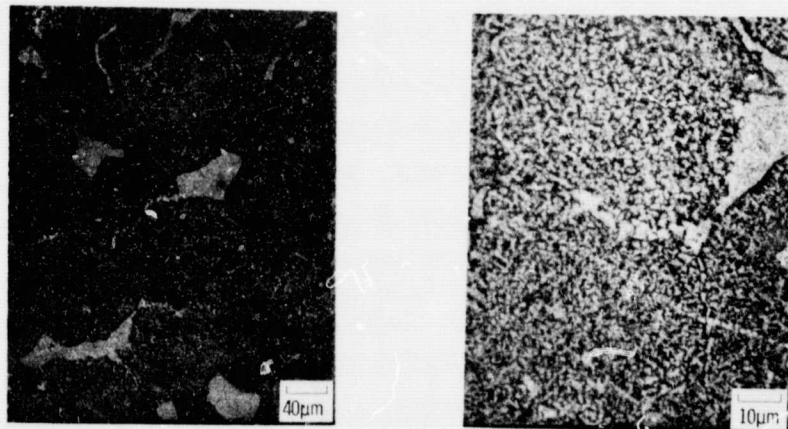


Figure 13. - VI A prealloyed powder product in condition A.

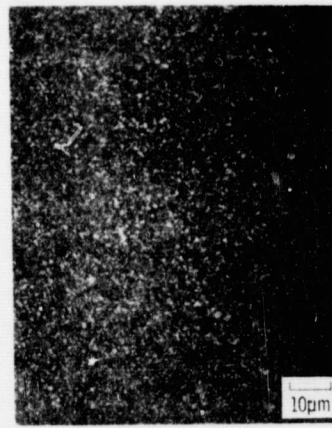
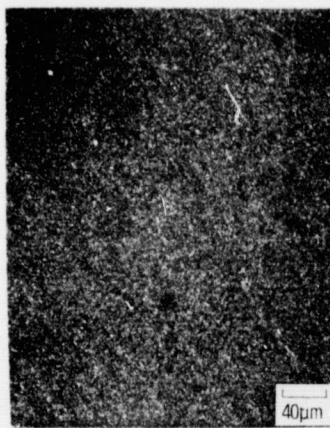


Figure 14. - VI A prealloyed powder product in condition B.

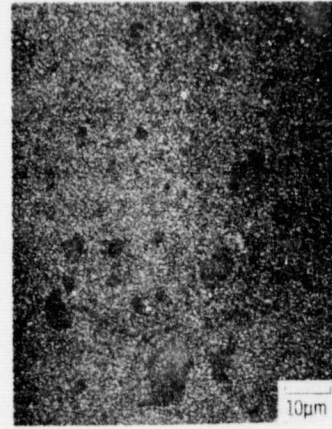
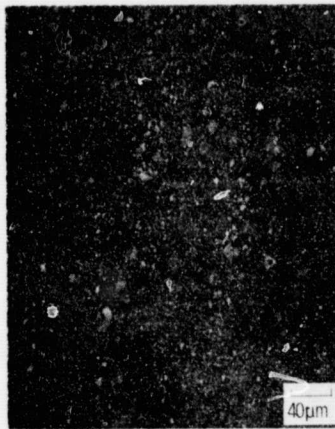


Figure 15. - VI A prealloyed powder product in condition C

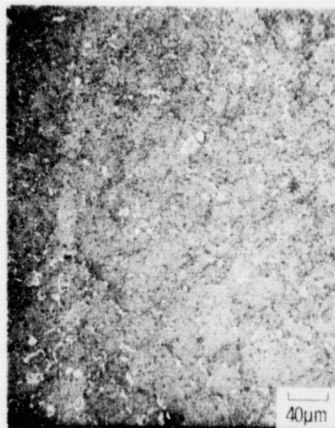


Figure 16. - VI A prealloyed powder product in condition D.

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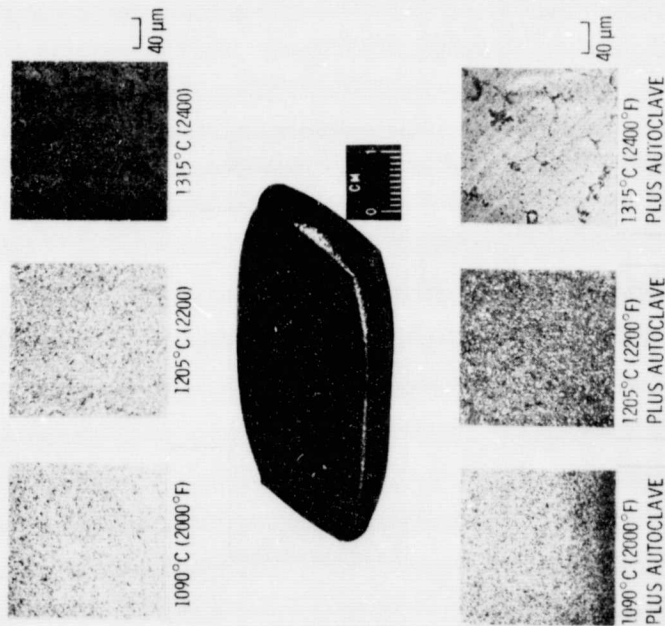


Figure 17. - VI A Extruded bar deformed by compression gradient heat treated for 1 hour with and without autoclaving.

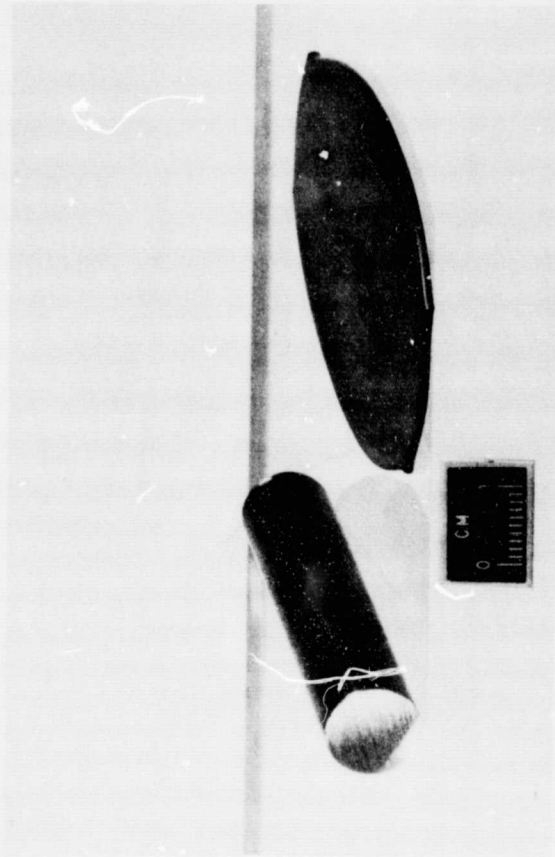


Figure 18. - Single step super plastic deformation of extruded VI A power product, (reduced 87% in thickness).

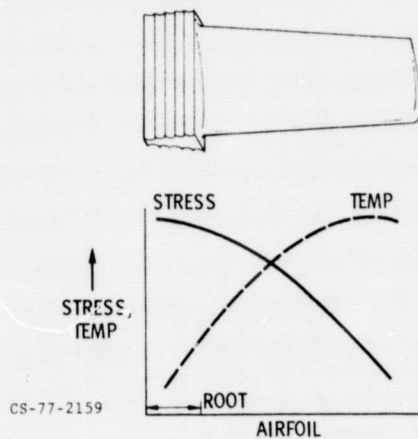


Figure 19. - Generalized stress-temperature distribution for advanced turbine blade.

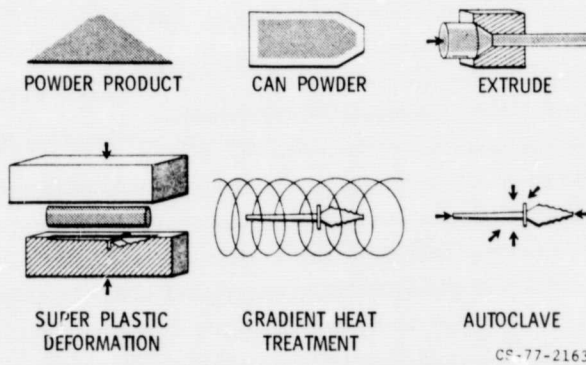


Figure 20. - Diagram of prealloyed powder product blade fabrication process involving autoclave heat treating for property control.